INVESTIGATION INTO THE FILTERING QUALITY OF RAW SUGAR

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Abstract

In response to occasional complaints from customers, attention has focussed on the filtration behaviour of raw sugars. Offending sugars, together with several others, were extensively analysed to establish the probable causes of the problem. Parameters correlating most strongly with poor filterability were turbidity and soluble phosphate (r = -0,83; n = 32) and (r = -0,76; n = 32) respectively. Starch (r = -0,67; n = 32), suspended solids (r = -0,66; n = 32), gums (r = -0,60; n = 32) and dextran (r = -0,50; n = 32) showed lesser correlations. When ranked according to phosphate levels, all the complaint sugars with the exception of one shipment were grouped at the top.

Introduction

To the refiner the filterability of raw melt is important because it influences refinery throughput. Processing of good quality raw sugars results in considerable savings in the refinery operation. Most raw sugar manufacturers are willing to do their utmost to provide sugar of acceptable quality as long as there are no great deviations from the normal methods employed in the manufacture of raw sugar. The findings presented here, although not novel, reflect the current situation with regards to certain quality parameters, in particular filterability, associated with South African raw sugar. They are certain to stimulate debate as to whether wholesale changes need to be made to the process currently employed in raw sugar production, or whether it is simply sufficient to ensure good process control with existing methods. Changes in clarifier design or a move to saccharate liming or sulphitation may effect the desired changes in sugar quality. A change in liming procedure would not involve considerable expense.

Based on results to-date it is evident that poor filterability is a sporadic problem. It is not clear, however, whether it is related to particular mills or is a consequence of seasonal or geographic influences. For a complete picture one would also require an investigation into the effects which cane variety and harvest techniques have. The investigations into these issues present a complex task made even more difficult by the fact that it is in all likelihood no one single factor that would impact on raw sugar filterability but rather a combination of several factors. Many of these factors are beyond the control of the mill which must therefore develop procedures to cope with the problem or optimise existing procedures.

The investigation into the filterability of raw sugar was initiated as a result of occasional complaints received from overseas customers. The complaints originated from predominantly one source, a new customer, operating a carbonatation refinery. Although some of the observations resulting from the investigation are interesting in so far as they have highlighted potential deficiencies in sugar quality, it must be borne in mind that the aim was to provide a 'quick fix' to a problem. The work may be viewed as incomplete in terms of a thorough investigation into the filtration impeding impurities in raw sugar. Retained samples of the problem sugar were analysed together with a number of other shipment sugars selected in such a way that a cross-section of good and bad filtering sugars made up the database. In addition samples of Komati sugar which had been analysed for filterability by the South African Sugar Terminals (SAST), and exhibited above average results, and two sugars (Benchmark 1 and Benchmark 2) provided by the complainant, and said to have good filterability were included as benchmark samples. A local carbonatation refinery (Tongaat-Hulett Refinery) (HR) had experienced filterability problems with certain sugars drawn from the South African Sugar Terminals and these – samples 2, 4 and 7 – complete the sample set.

Several analyses are routinely performed either by the SAST or by the Sugar Milling Research Institute (SMRI) and these together with the analytical results for suspended solids, bagacillo, turbidity, pH, elemental analysis and phosphates (P_2O_5 ppm) are given in Table 1. The additional analytical parameters are some of those which have been reported in previous publications as having an impact on sugar filterability. All refiners do not employ the same method of melt clarification and a sugar that behaves well in a carbonatation refinery may not necessarily do so in a phosphatation refinery. How the individual impurities impact on filterability with particular reference to a carbonatation refinery is discussed later.

Experimental

Thirty-two sugars (unaffinated) were analysed for those parameters indicated in Table 1. The filterability test is routinely performed by SAST and is essentially the method outlined by Nicholson and Horsley (1956). Test methods for the other analyses were taken from the ICUMSA Methods Book and the Laboratory Manual for South African Sugar Factories (Anon, 1985). Dextran levels were measured using the Roberts (1982) method.

Table 1. Analytical results for raw sugar tested.

Ship No/Sample	Pol	Moisture %	ICUMSA colour	Reducing sugars %	Conductivity ash %	Starch ppm	Gums ppm	Dextran ppm	Suspended solids ppm	Bagacillo ppm	Turbidity @ 900 nm	pH	Ca ppm	Mg ppm	Fe ppm	Silica SiO_2 ppm	Phosphate P_2O5	Filterability %
1*	97,80	0,44	2 441	0,97	0,23	154	1 600	615	623	51	4,40	6,49	200	80	2,4	152	136	19
2*	99,30	0,11	1 975	0,14	0,16	169	1 549	981	175	44	4,10	7,05	163	80	6,4	128	130	29
3	99,29	0,10	1 798	0,21	0,14	84	850	401	129	65	3,10	6,97	116	40	5,2	56	120	44
4*	99,30	0,11	2 188	0,15	0,18	158	1 415	822	167	44	2,70	6,91	120	40	4,0	88	120	50
5*	98,76	0,24	2 090	0,46	0,19	138	1 150	314	328	140	3,90	6,81	135	120	4,0	144	106	26
6*	98,78	0,25	1 923	0,47	0,15	131	1 800	490	161	41	2,30	6,82	168	40	3,6	96	104	48
7*	99,32	0,12	2 260	0,13	0,16	130	1 560	820	262	64	2,60	6,93	144	40	4,8	88	100	47
8*	98,74	0,23	1 758	0,55	0,14	80	900	447	174	49	2,70	6,78	136	40	4,4	128	99	51
9*	97,80	0,49	2 055	1,03	0,22	133	950	223	154	95	2,30	5,92	320	40	2,0	112	96	45
10	99,40	0,09	1 633	0,18	0,14	78	850	478	142	48	2,00	6,91	94	36	2,4	40	96	49
11	99,40	0,09	1 398	0,15	0,13	74	800	410	109	40	1,70	6,88	94	36	1,4	48	88	54
12	99,04	0,18	1 575	0,35	0,15	96	950	409	128	64	1,90	6,78	116	40	2,8	56	88	46
13	99,05	0,16	1 680	0,39	0,15	98	850	404	154	82	2,10	6,74	108	40	3,2	128	85	39
14	99,09	0,13	1 628	0,31	0,15	88	750	330	151	66	2,20	6,77	112	80	3,2	128	85	41
15	99,03	0,17	1 728	0,35	0,14	81	900	428	144	50	2,30	6,81	120	40	3,2	168	85	61
16	99,29	0,17	1 618	0,17	0,19	110	1 100	289	100	60	2,00	6,79	116	40	2,8	80	80	58
17	97,76	0,39	1 860	1,11	0,17	92	850	304	140	75	2,10	6,50	158	40	2,4	152	80	57
18	98,81	0,23	1 668	0,47	0,17	113	950	270	114	58	1,80	6,66	110	40	1,6	72	80	51
19	99,07	0,16	1 595	0,28	0,16	108	1 250	208	150	56	2,20	6,90	108	80	2,8	176	72	45
20	99,31	0,09	1 380	0,20	0,13	85	700	370	114	64	1,40	6,76	84	40	1,6	48	72	59
21	97,75	0,43	1 728	1,12	0,19	90	800	343	107	74	2,20	6,37	138	48	1,6	40	72	60
22	99,11	0,14	1 708	0,32	0,15	82	900	482	155	63	2,20	6,84	112	40	3,2	160	72	47
23	97,77	0,39	1 980	1,08	0,21	125	1 050	310	145	68	1,90	6,47	126	80	2,4	152	72	53
24	97,80	0,45	1 793	1,15	0,21	121	1 000	472	176	108	1,90	6,51	168	80	2,4	176	67	43
25	97,83	0,41	1 702	1,14	0,19	128	850	508	121	47	1,40	6,40	123	40	1,6	56	64	55
26	98,73	0,29	1 430	0,55	0,16	102	1 000	405	140	44	1,70	6,48	122	40	2,0	32	64	52
Komati WE16/4	99,25	0,15	1 600	0,16	0,19	43	630	190	77	29	1,70	6,53	98	40	2,0	144	60	66
Komati WE20/4	99,35	0,11	1 200	0,16	0,16	36	300	198	51	28	0,98	6,52	106	40	1,2	80	56	90
27*	98,81	0,22	1 503	0,58	0,16	115	800	400	81	44	1,50	6,63	103	40	1,6	176	51	52
Komati WE13/4	99,48	0,12	1 044	0,12	0,14	42	760	286	153	30	1,50	6,84	80	24	2,8	72	50	73
Benchmark 1	99,01	0,22	2 130	0,20	0,22	88	520	245	116	85	1,10	6,11	154	80	2,0	136	20	80
Benchmark 2	99,48	0,07	1 230	0,12	0,15	111	840	300	80	12	1,10	6,34	128	40	3,2	88	14	70
Average	98,83	0,22	1 728	0,46	0,17	103	974	411	157	59	1,96	6,66	131	51	2,3	106	81	52

Results

Pol, moisture, colour, reducing sugars, pH and conductivity ash results are not discussed but presented in Table 1 so that one can categorise the sugars analysed. The other parameters are discussed individually.

Starch

Starch has been recognised as an impurity which can severely impede filtration performance, particularly in a carbonatation refinery. A level of 150 ppm or less is generally considered to be non-troublesome, and the industry values depicted in Figure 1 show that this impurity has been kept to acceptable levels. The starch levels in shipment No. 1 and two complaint sugars received by HR exceed the 150 ppm level. Linear correlation coefficients given as a matrix of r values in Table 2 indicate that on the basis of the laboratory filterability test there is a strong correlation with starch levels (r = -0,67; n = 32). The linear regression is given in Figure 2. It is interesting to note that the three Komati sugars have very low levels of starch.

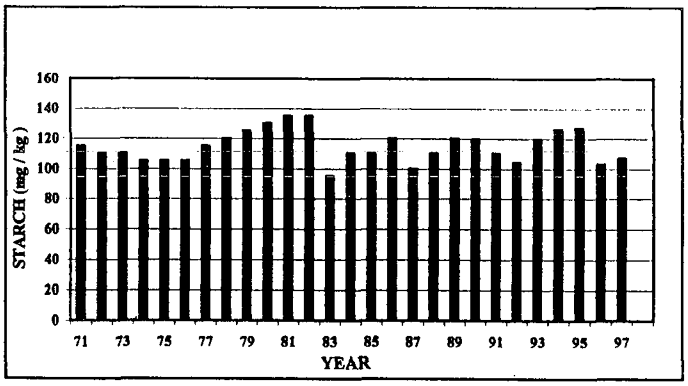

Figure 1. Starch levels in very high pol (VHP) sugar received by the South African Sugar Terminals.

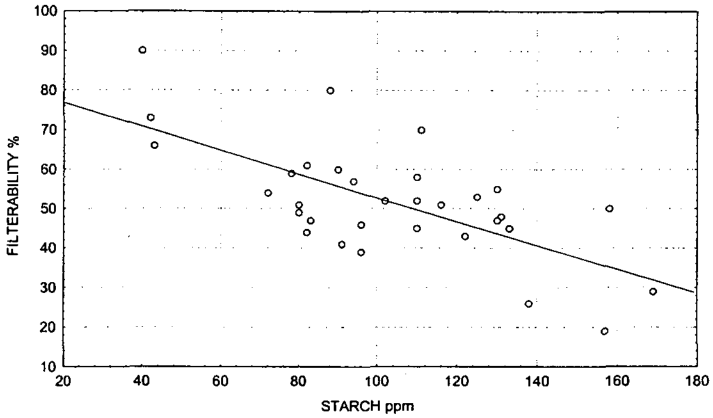

Figure 2. The linear regression for filterability versus starch (r = -0,67; n = 32).

Gums

Gums are essentially all the polysaccharide type materials which are precipitated by alcohol. Six of the nine complaint sugars (those marked with an asterisk in Table 1) had above average levels of gums ranging from 1 150 to 1 800, i.e. 176 to 826 ppm above the average of 974 ppm.

Dextran

Dextran was measured by the Roberts method and from Table 1 it can be seen that six of the complaint sugars exhibit levels ranging from 447 to 981 ppm, i.e. 36 to 570 ppm above the average of 411 ppm. The industry dextran levels for sugars received by SAST, for the last twelve years, are given in Figure 3. It can be seen that the trend over the past five years is an upward one.

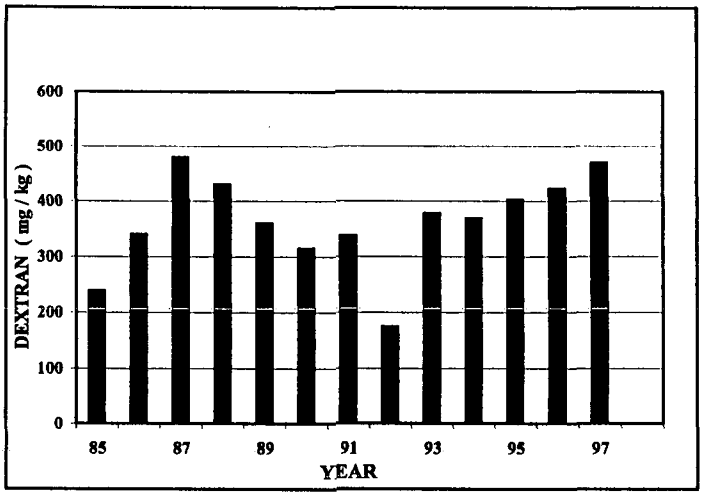

Figure 3. Dextran levels in VHP sugar received by the South African Sugar Terminals.

Suspended solids

Suspended solids are widely considered as the greatest contributor to filtration impedance. Shipment No. 1 has a suspended solids value of 623 ppm which is considerably higher than the average of 157 ppm. Shipment No. 5 and Sample 7, two further complaint samples, also have higher than average values of 328 and 262 ppm respectively. The linear correlation coefficient from Table 2 indicates that suspended solids are strongly correlated with laboratory filterability (r = -0,667; n = 32). When a logarithmic relationship is applied to the data the correlation is even more evident (r = 0,97; n = 32). The regression of filterability versus log (suspended solids) is given in Figure 4.

Bagacillo

Of the complaint sugars only the sample from shipment No. 5 has an excessively high bagacillo level of 140 ppm compared to the average of 59 ppm. When compared to the Benchmark 2 sample, however, all the South African sugars exhibit high bagacillo levels and this was evident from inspection of the membranes used for the analyses of suspended solids.

Turbidity

Many parameters analysed contributed to the turbidity measurement and it is therefore not unexpected that there should be a strong correlation between turbidity and laboratory filterability (r = -0,83; n = 32).

Table 2. Correlation coefficients (r) for all variables tested.

Variable	Filterability	Pol	Moisture	Reducing sugars	Ash	Colour	Starch	Dextran	Gums	Suspended solids	Turbidity	Bagacillo	Ca	Mg	Fe	Si	P_2O_5
Filterability	1,00	–	–	–	–	-0,59	-0,67	-0,50	-0,60	-0,66	-0,83	–	–	-0,47	-0,46	–	-0,76
Pol	–	1,00	-0,98	-0,98	-0,77	–	–	–	–	–	–	–	-0,58	–	–	–	–
Moisture	–	-0,98	1,00	0,94	0,78	–	–	–	–	–	–	–	0,65	–	–	–	–
Reducing sugars	–	-0,98	0,94	1,00	0,72	–	–	–	–	–	–	–	0,53	–	–	–	–
Ash	–	-0,77	0,78	0,72	1,00	0,65	0,49	–	–	–	–	0,49	0,63	0,49	–	–	–
Colour	-0,59	–	–	–	0,65	1,00	0,69	0,46	0,57	0,62	0,67	0,48	0,57	0,48	–	–	0,57
Starch	-0,67	–	–	–	0,49	0,69	1,00	0,60	0,75	0,49	0,59	–	0,52	0,45	–	–	0,49
Dextran	-0,50	–	–	–	–	0,46	0,6	1,00	0,66	–	0,56	–	–	–	0,63	–	0,60
Gums	-0,60	–	–	–	–	0,57	0,75	0,66	1,00	0,57	0,66	–	–	–	0,53	–	0,66
Suspended Solids	-0,66	–	–	–	–	0,62	0,49	–	0,57	1,00	0,75	–	–	0,47	–	–	0,56
Turbidity	-0,83	–	–	–	–	0,67	0,59	0,56	0,66	0,75	1,00	–	–	0,48	0,67	–	0,86
Bagacillo	–	–	–	–	0,49	0,48	–	–	–	–	–	1,00	–	0,59	–	–	–
Ca	–	-0,58	0,65	0,53	0,63	0,57	0,52	–	–	–	–	–	1,00	–	–	–	–
Mg	-0,47	–	–	–	0,49	0,48	0,45	–	–	0,47	0,48	0,59	–	1,00	–	0,51	–
Fe	-0,46	–	–	–	–	–	–	0,63	0,53	–	0,67	–	–	–	1,00	–	0,55
Si	–	–	–	–	–	–	–	–	–	–	–	–	–	0,51	–	1,00	–

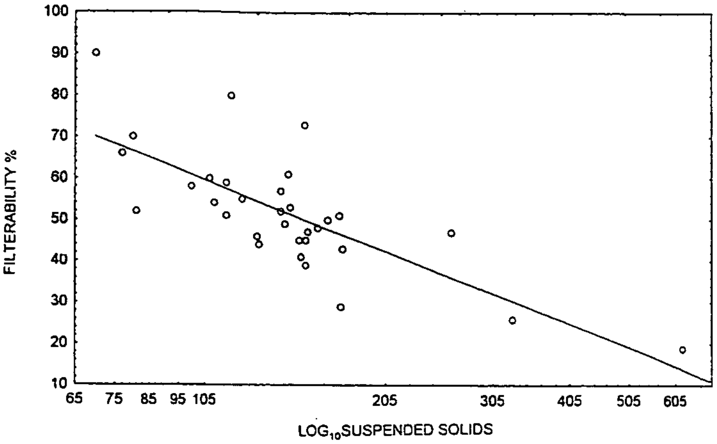

Figure 4. The linear regression for filterability versus log (suspended solids) (r = 0,97; n = 32).

Elemental analysis (Ca, Mg, Fe, Si)

No startling correlations were found for any of these parameters with the laboratory filterability. Surprisingly, however, no significant correlation was found between calcium and phosphate.

Phosphate

Phosphates are determined by a colourimetric method and refer here to soluble phosphate. Of all parameters analysed, the strong correlation (r = -0,76; n = 32) of phosphate with laboratory filterability, given in Table 2, was probably the least expected. The two Benchmark sugars analysed have phosphate levels of 14 and 20 ppm, well below the average of 81 ppm and indeed all of the South African raw sugars analysed. The linear regression is shown in Figure 5.

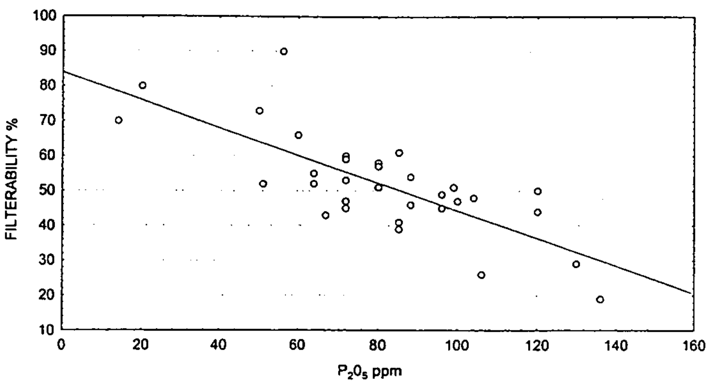

Figure 5. Linear regression for filterability versus phosphate (r = -0,67; n = 32).

Discussion

The results are in agreement with Hidi and McCowage (1984), who believe that filtration impedance is the result of several factors rather than one single one. The complaint raw sugar shipments were found to have higher than average levels of suspended solids, starch and phosphates. Although these are not necessarily the only factors likely to cause filtration impedance, bringing them down to acceptable levels (whatever those might be set at) would probably be a good starting point towards the improvement of raw sugar filterability and indeed quality. The origins of these filtration impeding impurities are not always clear nor are the mechanisms by which they cause filtration impedance. Many of the comments made here are taken from previous publications by other researchers and are intended to give some explanation.

Starch

Starch levels are dependent on cane maturity, cane variety, and the height at which the cane is topped. Starch is not considered to be a problem in the South African industry since the introduction of α-amylase enzymes and then diffusion. Not all factories use enzymes, particularly those with diffusers. Some sugars with excessive starch may be delivered for export. It is acknowledged that this sugar is penalised but markets still have to be found. Starch consists of two fractions but it is the amylose fraction which is responsible for poor crystal agglomeration and the subsequent bad filterability of the calcium carbonate slurry (Murray *et al.* 1974). Many raw sugars containing starch in excess also contain phosphate in excess (Nicholson, 1960). The effect on filterability of one impurity may be enhanced by the presence of another (Davis, 1959). It may therefore be necessary to reduce levels of starch below 150 ppm where levels of phosphate are high. If there has been a change in the amylose:amylopectin ratio, for whatever reason (possibly a varietal change or an effect of diffusion) and the amylopectin fraction has increased, there may be increased levels of phosphate as a result of the protective colloid action of amylopectin.

Phosphate

High phosphate levels are generally associated with ineffective pH control (Davis, 1959). In a carbonatation refinery this would tend to reduce filterability as a result of the formation of fine calcium phosphate crystallites which block the interstices in the matrix formed by calcium carbonate aggregates. Where calcium carbonate agglomeration is hampered by starch this effect may be more pronounced as less calcium phosphate may be required to block the interstices. The protective colloid action of dextran may also result in higher phosphate levels in sugar. Dextran levels increase as a result of cane delays and are often associated with heavy rainfall (e.g. the floods of 1987 – Figure 1). The situation is made worse if temperatures are high and there may at times be a dextran 'epidemic'. As juices become more refractory the tendency would be to increase phosphate addition. In these circumstances the possibility exists that 'overdosing' with phosphates may not be countered by adequate liming due to poor pH control. The protective colloid action of dextran is not negated and will contribute to an already high phosphate level. Dosing with dextranase might be considered as a remedy but can only be practised where temperatures permit. Alternatively, and preferably, there should be a reduction in the delays between burning and crushing of cane.

Refractory juices may also invite increased dosage of polyacrylamide flocculants. Residual flocculants are known to have an impact on filterability (Crees *et al.* 1977). Unfortunately there are no methods to analyse for residual polyacrylamides in sugars. If residual flocculant is returned to the mixed juice in one of the process streams it also has the ability to behave as a protective colloid and increase phosphate levels. The choice of flocculant is important as it has been shown that, depending on the phosphate content of the juice, some are more effective than others (Shephard, 1980). In Table 1, in which the sugars have been ranked according to phosphate levels, all the complaint sugars are at the top with the exception of the shipment No. 27.

Suspended solids

As mentioned earlier, several researchers have found suspended solids to be one of the main contributors to poor filterability especially suspended solids with a particle size below 5 μm (Donovan and Lee, 1995). It has been shown that part of this size is made up of field soil particles (Devereux and Clarke, 1984). An increase of soil into the factory would occur following heavy rain especially if burning was incomplete or if there were delays resulting in dextran formation. Dextran would act as 'glue', adhering soil particles to the cane. Insoluble calcium phosphate carried over as part of the muds may be considered as part of the suspended solids and arises from floc damage due to turbulence or running the clarifier with mud levels too high.

Bagacillo

Although there does not appear to be a strong correlation between bagacillo and laboratory filtration tests, the analysis was included as the refiner indicated that raw sugars with high bagacillo levels had in the past caused filtration difficulties. If the refinery was used to receiving sugar like the Benchmark sugar with bagacillo levels of 12 ppm the observation could well be valid. Bagacillo in sugar is often the result of poor flashing or airborne bagacillo getting into open top A-crystallisers.

Conclusions

Work done in South Africa relating to the filterability of raw sugar is dated. Alexander (1957) found that starch, silica and waxes had the greatest effect on filterability. He stated that no correlation was found between phosphate and resistance to filtration and that this was possibly due to the fact that the sugars examined were from sulphitation mills and that with increasing amounts of defecation sugar being produced, phosphates may be found to have a more pronounced effect. Work done in the early seventies focused on the influence of starch and insoluble suspended matter (Murray, 1972). It would probably be wise to introduce phosphate analyses as routine indicators of sugar quality in the light of the findings of this present investigation.

It has become apparent that the measurement of suspended solids and turbidity may be useful in determining sugar quality with respect to filterability. Turbidity measurements are quick, require inexpensive equipment and could be performed by factory personnel. Decisions to release sugar to SAST could be made on the basis of these measurements and the SAST could be notified in advance of a consignment of substandard quality allowing appropriate action to be taken in handling this sugar.

The Australian industry when confronted with the problem of one of its mills producing sugar of low filterability conducted an investigation and found that some of the principles laid down for process control were being contravened (Davis, 1959). To rectify this, attention to the following details was recommended and probably provides the quickest, least expensive way to address the filtration problem:

- Phosphate content of mixed juice - this should be adjusted if necessary.
- Floc damage due to turbulence - liming should be deferred until after the heaters to delay the formation of most of the floc.
- Liming control - good control of pH is required to prevent inadequate liming resulting in the passage of soluble phosphate into the raw sugar. This is particularly important when processing refractory juices.
- Saccharate lime - studies show that saccharate lime improves clarification (Davis, 1959). The increased mud volumes associated with saccharate liming found by Scott (1988) may detract from the use of this procedure.
- Gas bubbles in the floc - it is necessary to ensure that flash tanks preceding clarifiers are operating correctly. Mud volumes would then be reduced. Operators would be less tempted to adopt liming procedures resulting in a damaged floc which would settle faster but give a less clear juice.
- Flow control - poor flow control would lead to floc damage and is probably the most likely source of carry-over.

Once production personnel are satisfied that all of the above details are in order and that no undue amounts of insoluble matter are passing into the clear juice, the matter of further improving filterability may be peculiar to a particular factory and require deeper investigation. Some of the points raised in the discussion section of this paper may be considered for investigation, together with the possibility of introducing syrup clarification.

As part of a continuing investigation into raw sugar filterability, samples have been obtained from all South African factories and will be analysed according to the same parameters used in this investigation. In this way it is hoped that any geographical, seasonal or factory influences will be highlighted, and establish the extent of the problem.

Acknowledgments

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